PRODUCTION OF CELLULOSE FIBER FROM OIL PALM FROND USING STEAM EXPLOSION METHOD

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“ I declare that this thesis is the result of my own research except as cited references. The thesis has not been accepted for any degree and is concurrently submitted in candidature of any degree.”

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DEDICATION

Dedicated to my beloved father, mother
Brothers and sisters…

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ABSTRACT

The world's largest producer and exporter of palm oil today is Malaysia, producing about 47% of the world's supply of palm oil. Indonesia is the second largest world producer of palm oil producing approximately 36% of world palm oil volume. Both nations are expanding their palm oil production capacity and the market continues to grow. It is estimate 26.2 million tonnes of oil palm fronds is treated as biomass in Malaysia. Palm frond consist huge amount of fiber while it can be transform because the fiber has cellulose in it. Majorly, fronds are taken from palm oil waste which is usually will be burn. Cellulose is the major constituent of paper, textiles and pharmaceutical products. This research is to prepare and produce the cellulose fiber from oil palm frond. Steam explosion pretreatment was conducted to enhance the accessibility of the oil palm frond lignocellulose. The combination of temperature and time known as the Log Ro in steam explosion process were used as the main parameter in this research. Several steps are involved in the preparation of cellulose such as steam explosion, alkaline treatment and bleaching. Temperature is use as parameter to achieve better product. Result shows that Log Ro giving contribution to the yield obtained. Steam exploded fiber giving yield in range 71 to 90%, alkaline extracted fiber : 70 – 90% yield and bleach fiber : 90 -95% yield from their raw materials.

ABSTRAK

Malaysia merupakan pengeksport terbesat kelapa sawit di seluruh dunia, Malaysia mengeluarkan kira – kira 47% daripada pengeluar minyak sawit dunia. Indonesia pula di tangga kedua dengan mengeluarkan kira – kira 36% minyak sawit. Kedua – dua negara mengembang pengeluaran minyak mereka dan permintaan pasaran semakin meningkat. Dianggarkan kira – kira 26.2 juta tan pelepah kelapa dibuang begitu sahaja di Malaysia. Pelepah kelapa mengandungi fiber yang dapat ditukarkan menjadi sellulosa kerana ia mengandunginya. Kebiasaannya, pelepah kelapa sawit ini di bakar begitu sahaja. Sellulosa merupakan bahan utama dalam pembuatan kertas, kain dan produk – produk farmasi. Kajian ini dijalankan untuk menghasilkan fiber sellulosa daripada pelepah kelapa sawit. Kaedah ledakan stim digunakan untuk meningkatkan keupayaan pelepah kelapa sawit menghasilkan lignosellulosa. Kombinasi penggunaan suhu dan masa tindakbalas yang dikenali Log Ro dalam ledakan stim digunakan sebagai parameter utama kajian. Beberapa kaedah digunakan dalam menghasilkan fiber sellulosa iaitu ledakan stim, pengekstrakan alkali dan proses pelunturan. Suhu digunakan sebagai parameter untuk menghasilkan produk yang lebih baik. Keputusan menunjukkan peratus hasil yang diperolehi dalam proses menghasil sellulosa fiber bergantung kepada Log Ro selepas ledakan stim. Hasil bagi ledakan stim adalah 71 – 90%, pengekstrakan alkali 70 – 90% dan pelunturan 90 – 95% daripada berat kering sample bahan mentah masing – masing.

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INTRODUCTION

1.1 BACKGROUND

Recycle lignocellulose waste of palm oil was a challenge to farmer and factory. Lignincellulose waste comprised 2 types which is farm waste and factory waste. This research is limit only for waste that are from palm oil estate or more specifically, palm oil frond.

Palms are one of the most well-known and extensively cultivated plant families. They have had an important role to humans throughout much of history. Many common products and foods are derived from palms, and palms are also widely used in landscaping for their exotic appearance making them one of the most economically important plants. In many historical cultures, palms were symbols for such ideas as victory, peace, and fertility. Today, palms remain a popular symbol for the tropics and vacations

The oil palms (Elaeis) comprise two species of the Arecaceae, or palm family. They are used in commercial agriculture in the production of palm oil. The African Oil Palm Elaeis guineensis is native to west Africa, occurring between Angola and Gambia, while the American Oil Palm Elaeis oleifera is native to tropical Central America and South America.

Mature trees are single-stemmed, and grow to 20 m tall. The leaves are pinnate, and reach between 3-5 m long. A young tree produces about 30 leaves a year. Established trees over 10 years produce about 20 leaves a year. The flowers are produced in dense clusters; each individual flower is small, with three sepals and three petals. The fruit takes five to six months to mature from pollination to maturity; it comprises an oily, fleshy outer layer (the pericarp), with a single seed (kernel), also rich in oil. Unlike other relatives, the oil palm does not produce offshoots; propagation is by sowing the seeds.

Oil palms are grown for their clusters of fruit, which can weigh 40-50 kg. Upon harvest, the drupe, pericarp and seeds are used for production of soap and edible vegetable oil; different grades of oil quality are obtained from the pericarp and the kernel, with the pericarp oil used mainly for cooking oil, and the kernel oil used in processed foods.

For each hectare of oil palm, which is harvested year-round, the annual production averages 10 tonnes of fruit, which yields 3,000 kg of pericarp oil, and 750 kg of seed kernels, which yield 250 kg of high quality palm kernel oil as well as 500 kg of kernel meal. The meal is used to feed livestock. Some varieties have even higher productivities which has led to their consideration for producing the vegetable oil needed for biodiesel.

The African oil palm was introduced to Sumatra and the Malaya area in the early 1900s; many of the largest plantations of oil palms are now in this area, with Malaysia growing over 20,000 square kilometers. Malaysia claims that in 1995 it was the world's largest producer with 51% of world production. Palm oil and its fractions are practical and attractive choice for importers and food manufacturers, especially in 3rd world countries due to its price competitiveness, year-round supply, diversity and versatility for edible and non-edible applications (Mohamad Husin *et al.*, 1986).

The oil palm industry in Malaysia started 80 years ago in a modest way. Today it is the largest in agricultural plantation sector, exceeding rubber plantation by more than double in area planted.

In terms of hectare, the total area under oil palm cultivation is over 2.65 million hectares, producing over 8 million tonnes of oil annually. The oil consists of only

10% of the total biomes produced in the plantation. The remainder consists of huge amount of lignocellulosic materials such as oil palm fronds, trunks and empty fruit bunches. The projection figures of these residues are as follows:

- 7.0 million tonnes of oil palm trunks
- 26.2 million tonnes of oil palm fronds
- 23% of Empty Fruit Bunch (EFB) per tonne of Fresh Fruit Bunch (FFB) processed in oil palm mill

Based on the above figures, Malaysia therefore has a great potential in turning its abundant supply of oil palm industry by-products into value-added products (www.aseansec.org/**7**011.htm).

Under the present scenario, Malaysia can no longer remain idle and complacent in its position as the top grower and supplier of palm oil. In view of the escalating challenge posed by the other oil producing countries, Malaysia has to change its objective of being a world producer of palm oil to amongst others a leader in converting biomass waste into value-added products. Malaysia has therefore to seriously resort in aggressive R&D to support its ambition.

Before embarked into identifying the parameters affecting the overall quality of the fiber, let turn to look into the current utilization of these fibers. Recent report shows that the monocarp fiber and shell are used as boiler to produce steam and to generate power. Whereas, empty fruit bunches are mainly incinerated to produce bunch ash to be distributed back to the field as fertilizer (www.freepatentsonline.com/ 4352341.htm).

The conventional method of burning these residues often create environmental problems in that it generates severe air pollution and is prohibited by the Environment Protection Act. In abiding by the regulations, these residues are becoming expensive to dispose. Nevertheless, looking on the brighter side of things, extensive research has provided us with an alternative way of optimizing the usage of oil palm residues fiber based into value-added product.

1.2 THE OBJECTIVES OF THE RESEARCH.

There are 2 objectives of the research which are to:-

i. Examine correlation between time and temperature in Reaction Ordinate (Ro).
ii. To produce cellulose from waste biomass cellulosic material, oil palm frond

1.3 THE SCOPES OF RESEARCH

There are 3 scopes of the research are to:

i. Use oil palm frond as alternative material to create high quality cellulose.
ii. The influence of temperature and time on the steam explosion method in producing cellulose fiber from cellulosic material (oil palm frond).
iii. The cellulose fiber shall be the main raw material input for pulp, paper and medical product.

1.4 PROBLEM STATEMENT

In the world today, biomass is became a massive problem to many country due to its causes to environment. Many studies have been made by several scientist but not all them can solve the problem of biomass. Utilization of waste material such as palm frond also being studied. The race for producing biodegradable products has increase tremendously. Different approaches have been attempted to use biomass as natural biopolymer for production of biodegradable plastics. It is almost 26.2 million tonnes of oil palm fronds in Malaysia according to MARDI. This biomass has been the main source cellulose fiber. This research is important because of Malaysia total net of importing cellulose is more than RM300 million per year and keep increasing.

So, this research will help palm oil sector to solve the biomass problem of oil palm frond and turn them into valuable product which is cellulose fiber.

CHAPTER 2

LITERATURE REVIEW

2.1 OIL PALM

2.1.1 The tree, history, distribution and habitat.

The oil palm is a tropical palm tree. There are two species of oil palm, the better known one is the one originating from Guinea, Africa and was first illustrated by Nicholaas Jacquin in 1763, hence its name, Elaeis guineensis Jacq.

Figure 2.1 The Fruit Bunch

The fruit is reddish about the size of a large plum and grows in large bunches. A bunch of fruits can weigh between 10 to 40 kilograms each. Each fruit contains a single seed (the palm kernel) surrounded by a soft oily pulp. Oil is extracted from both the pulp of the fruit (palm oil, an edible oil) and the kernel (palm kernel oil, used mainly for soap manufacture). For every 100 kilograms of fruit bunches, typically 22 kilograms of palm oil and 1.6 kilograms of palm kernel oil can be extracted (www.en.Wikipedia.org/wiki/ Oil_palm).

The high productivity of the oil palm at producing oil (as high as 7,250 liters per hectare per year) has made it the prime source of vegetable oil for many tropical countries. It is also likely to be used for producing the necessary vegetable oil for biodiesel, an example being a planned refinery Darwin, Australia which will import the palm oil from Indonesia and Malaysia.

The oil palm originated in West Africa but has since been planted successfully in tropical regions within 20 degrees of the equator. There is evidence of palm oil use in Ancient Egypt.

Figure 2.2 Plantation

In the Republic of the Congo, or Congo Brazzaville, precisely in the Northern part, not far from Ouesso, local people produce this oil by hand. They harvest the fruit, boil it to let the water part evaporate, then they press what its left in order to collect the reddish, orange colored oil.

The world's largest producer and exporter of palm oil today is Malaysia, producing about 47% of the world's supply of palm oil. Indonesia is the second largest world producer of palm oil producing approximately 36% of world palm oil volume. Both nations are expanding their palm oil production capacity and the market continues to grow.

Worldwide palm oil production during the 2005-2006 growing season was 39.8 million metric tons, of which 4.3 million tons was in the form of palm kernel oil. It is thus by far the most widely-produced tropical oil, and constitutes thirty-four percent of total edible oil production worldwide. Oil palm is one of the most abundant, unutilised waste biomass from plantation in South-Asia (www.en.wikipedia.org/wiki/Oil_palm**).**

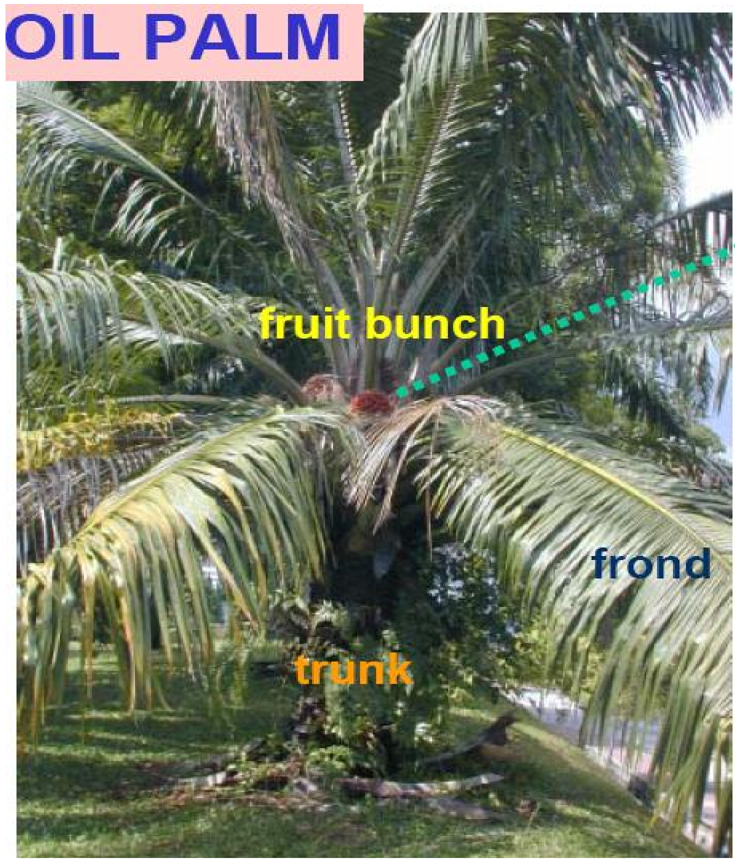

Figure 2.3 The tree

2.1.2 Palm Frond

A palm branch (or palm frond or palm stem), usually refers to the leaves of the Arecaceae (sometimes known by the names Palmae).

The palm branch was a symbol of triumph and victory in pre-Christian times. The Romans rewarded champions of the games and celebrated military successes with palm branches. Jews followed a similar tradition of carrying palm branches during festive times. Early Christians used the palm branch to symbolize the victory of the faithful over enemies of the soul, as in the Palm Sunday festival celebrating the triumphal entry of Jesus into Jerusalem.

In Judaism, the date palm represents peace and plenty, and is one of the Four Species (Lulav) used in the daily prayers on the feast of Sukkot. It is bound together with the hadass (myrtle), and aravah (willow) The palm may also symbolize the Tree of Life in Kabbalah. While in Islam, Muhammad is said to have built his home out of palm, and the palm symbolizes rest and hospitality in many cultures of the Middle East. The first muezzin climbed palm trees to call the faithful to prayer, from which the minaret developed.

The cell walls of OPF are composed mainly of cellulose, hemicelluloses and lignins. In addition to those three main components, several percent of ash and extractives are included. Among those components, cellulose and hemicelluloses are polysaccharides, which are easily decomposed and metabolized by wood-rot fungi. In the constituent analysis of wood, when lignin is selectively removed, what is obtained is holocellulose. Holocellulose can be thought of as the total of cellulose and all hemicelluloses (KOZUMAYosei *et al.*,).

Fiber property	Oil Palm Frond
Mean fiber length (mm)	
Arithmetic	0.59
Length weighted	1.13
Weight weighted	1.54
Coarness (mg/m)	0.098
Fiber dimensions(μm)	
Fiber diameter (D)	19.6
Lumen Width (L)	11.66
Cell wall thickness (T)	3.97

* Source : Law and Jiang (2001)

Table 2.1 Fiber Characteristics of Oil Palm Fronds

Lignin of oil palm frond was characterized by the presence of significant amounts of esterified p-hydroxybenzoic acid together with small amounts of etherified ***p***-hydroxybenzoic acid. Vanillic and siringic acids were esterified or etherified to lignin. Some extents of these ester bonds and *beta*-O-4 interunit linkages of lignin were cleaved during steam explosion, in addition to great condensation of guaiacyl nuclei, as revealed by ^{1}H- and ^{13}C-NMR spectra of isolated lignins from the steam exploded pulps, of which yields were quite high, suggesting that lignin has been released from other wall polymers. Wall polysaccharides of oil palm frond are composed of cellulose and significantly high concentration of arabinoxylan, which produced great abundance 5-hydroxymethyl-furfural and furfural during steam explosion, respectively and even hot pressing at 125 ^{0}C to prepare binderless boards. It is suggested that released lignin and furfural derivatives generated during steam explosion contribute to self-binding of the steam exploded pulps. However, severe conditions of steam explosion caused great damages in lignin macromolecules, and gave poor quality of binderless board.

Cmponent	Oil Palm Frond
Lignin	15.2
Holocellulose	82.2
Alpha cellulose	47.6
Ash	0.7
Polysaccharide Composition	
Arabinose	1.5
Mannose	2.2
Galactose	0.9
Glucose	66.6
Xylose	28.9

* Source : Law and Jiang (2001)

Table 2.2 Chemical Compositions of Oil Palm Fronds

Bioconversion of lignocellulosic waste materials to chemicals and fuels are receiving interest as they are low cost, renewable and widespread in nature. Malaysia is well known for its potential in renewable resources such as oil palm waste, sugar cane bagasse and rice straw. At present Malaysia is the largest exporter of palm oil in the international market. In the process of extraction of palm oil from oil palm fruit, a lignocellulosic material oil palm empty fruit bunch (OPEFB) and palm frond is generated as a waste product. Approximately 15 million tons of OPEFB and frond biomass waste is generated annually throughout Malaysia by palm oil mills. In practice this biomass is burned in incineratorsby palm oil mills which not only creates environmental pollution problems in nearby localities but also it offers limited value to the industry. The OPEFB and frond biomass contains cellulose, hemicellulose and lignin. It is estimated that OPEFB biomass is comprisedof 24% xylan, a sugar polymer made of pentose sugar xylose. This xylose can be used as substrate for production of a wide variety of compounds by chemical and biochemical processes (Azemi *et al*., 1996).

2.1.3 Conservation Status

Optimal plant density is 58 trees/acre with triangular patterns about 30 ft apart. During the first 3 years, little or no fruit is obtained and plantations are often intercropped with staple crops. Pruning and Training - none, old leaves are pruned off to facilitate access to the bunch at harvest. When palms reach heights of 20-30 ft, they become difficult to harvest, and are often injected with an herbicide to kill them or bulldozed down. New trees are planted among the dead and rotting trunks

While it is still unclear as to the status of the oil-palm plantations in central Borneo, the Friends of the Earth report lays out a set of recommendations for making better ecological and economic use of the rainforests of Kalimantan. If the government does intend to carry on with the proposed project, then it should first focus on increasing productivity in existing plantations, not on clearing new land for palm..

This can be done using improved seeds and doing a better job of adopting harvesting practices from other parts of the world, while encouraging replanting of abandoned and degraded plantations.

2.1.4 Harvesting of the frond

It is an agricultural plant, which originates from West Africa and cultivated in Malaysia for its oil producing fruit. Besides palm oil, the industry also generates massive amounts of lignocellulosic residues such as trunks, fronds and the empty fruit bunches (EFB), with an estimated amount of 30 million tonnes (MPOB, 2001). The suitability of this abundant, inexpensive and renewable raw material for papermaking resource has been explored using a variety of pulping methods. Majorly, fronds are taken from palm oil waste which is usually will be burn.

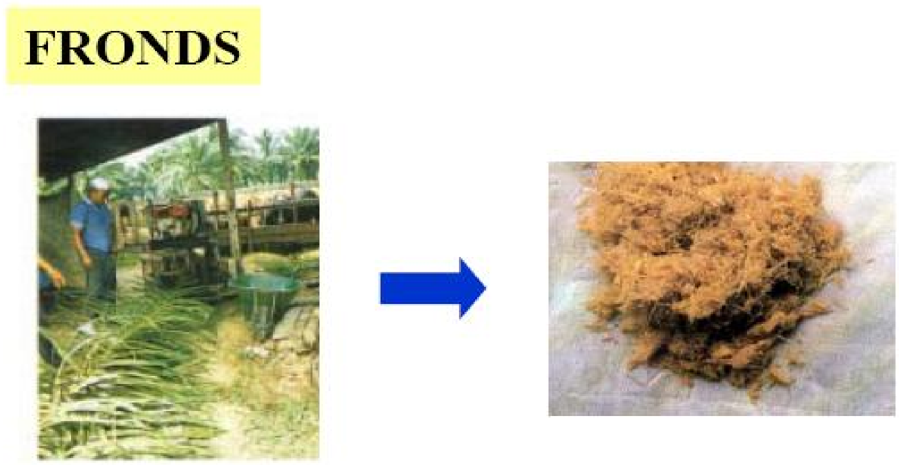

Figure 2.4 From fronds to fiber

2.2 FROND PROPERTIES, CHEMICAL COMPOSITION

Component	Oil palm fronds	EFB	Hardwood	Softwood
Lignin (%)	20.5	17.2	25.2	21-37
Holocellulose (%)	83.5	70	72.4	60-80
α-Cellulose (%)	49.8	42.7	44.7	31-60
Alcohol–benzene extractives (%)	1.4			
Ash (%)	0.7		< 1	< 1
Alkali solubes (%)		17.2	13.6	
Pentosans (%)		27.3	12.9	

* Source : Law and Jiang (2001)

Table 2.3 Chemical Compositions of Oil Palm

2.2.1 Cellulose

Cellulose is an organic compound with the formula (C6H10O5)n. It is a structural polysaccharide derived from beta-glucose. Cellulose is the primary structural component of green plants. The primary cell wall of green plants is made of cellulose; acetic acid bacteria are also known to synthesize cellulose, as well as many forms of algae, and the oomycetes. Cellulose was discovered and isolated in the mid-nineteenth century by the French chemist Anselme Payen with an estimated annual production of 1.5x109 Tonnes.

Some animals, particularly ruminants and termites, can digest cellulose with the help of symbiotic micro-organisms - see methanogen. Cellulose is not digestible by humans and is often referred to as 'dietary fiber' or 'roughage', acting as a hydrophilic bulking agent for feces.

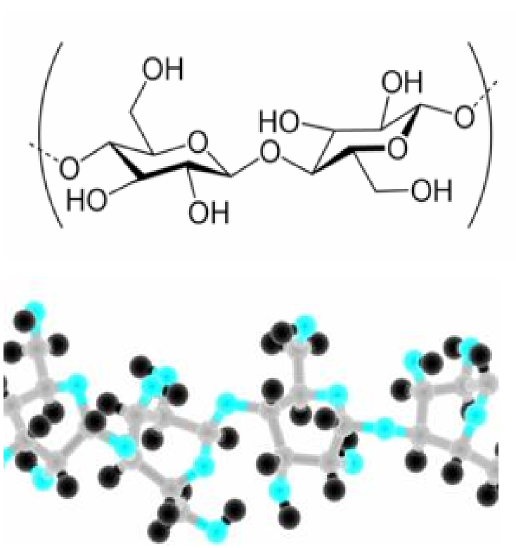

Figure 2.5 Molecule Structure of Cellulose

Cellulose is derived from (β-glucose), which condense through β(1→4)-glycosidic bonds. This linkage motif contrasts with that for α(1→4)-glycosidic bonds present in starch and other carbohydrates. Cellulose is a straight chain polymer: unlike starch, no coiling occurs, and the molecule adopts an extended rod-like conformation. In microfibrils, the multiple hydroxyl groups on the glucose residues hydrogen bond with each other, holding the chains firmly together and